System for Measuring Spectral Distribution of Normal Emissivities of Metals in Direct Current Heating¹

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ABSTRACT

A system for measuring time variations of the normal spectral emissivity ranging from $0.55\mu m$ to $5.3\mu m$ was developed and applied to metal specimens in vacuum and oxidizing environments in the temperature range from $780^{\circ}C$ to $1200^{\circ}C$. The specimen was heated at high temperatures by direct current pass in a vacuum chamber, and the surface oxidation was controlled by evacuation and introduction of oxidizing gas. The specimen temperature was measured by a single-band $(0.9\mu m)$ radiation thermometer viewing a cavity formed in the specimen from the rear side. The front surface of the specimen was observed by a multiband (112 wavelengths) radiation thermometer to measure the normal spectral emissivity.

The effective normal spectral emissivity of the specimen cavity was evaluated to be 0.94±0.05 at the wavelength of 0.9µm in comparison with a metal tube having a small blackbody hole on the rear. The measurement accuracy of the normal spectral emissivity by the system was estimated to be 5% to 10% of the emissivity value in most of the interesting ranges of emissivities, temperatures and wavelengths.

KEY WORDS: high temperature; metal; spectral emissivity; oxidation; radiation thermometer.

1. INTRODUCTION

Knowing the spectral emissivity of a target is indispensable for measuring the temperature by thermal radiation. However, it can easily change due to physical and chemical conditions of the surface, especially to surface oxidation for metals. Changes in the spectral emissivity of metals have been investigated during oxidation processes at selected wavelengths [1,2,3], and waving of the spectral emissivity has been observed for some metals as surface oxidation progresses. It is understood from those investigations that accurate temperature measurements of metal surfaces need a new technique of radiation thermometry rather than conventional single-band or two-band radiation thermometers.

Recent development of electronics is making easier realizing high performance with multiband radiation thermometers [4]. Spectral radiance measurements covering a wide wavelength range at continuous wavelengths for a short scanning period are now available by improvements of infrared array detectors and signal processors. New multiband radiation thermometers seem promising to have a possibility of emissivity compensation for radiation thermometry of metals in oxidation processes.

Makino has developed a high-speed spectrometer [5], and measured time variations of the spectral reflectivity of metals in an oxidizing environment at high temperatures [6]. The work suggested the possibility of measuring time variations of the spectral emissivity for metal surfaces in oxidation processes. The spectral emissivity may lead to more direct contribution than the spectral reflectivity to radiation thermometry.

This paper describes a system for measuring spectral distribution of normal emissivities of metals in a wide wavelength range for a short period in various environmental conditions including oxidation. An attention was paid to increasing the measurement efficiency because it was aimed to measure a lot of specimens for building a normal spectral emissivity database consisting of a wide variety of metals.

2. Measurement system

The measurement system consisted of specimens, a multiband radiation thermometer for the specimen radiance measurement, a single-band radiation thermometer for the specimen temperature measurement, and a specimen heating/environmental control system.

2.1. Measurement system

Figure 1 shows a schematic of the measurement system. The front surface of the specimen heated by direct current pass was observed at the middle of the specimen by the multiband radiation thermometer to measure the spectral radiance. The specimen temperature was measured from the rear by the single-band $(0.9\mu\text{m})$ radiation thermometer. The normal spectral emissivity of the specimen was derived from the temperature measured by the single-band radiation thermometer and the spectral radiance on the specimen front surface at the normal direction measured by the multiband radiation thermometer.

Surface oxidation of the specimen was controlled by evacuation and introduction of oxidizing gases into a vacuum chamber. A change of atmospheric absorption due to water vapor and carbon dioxide along the optical path of the multiband radiation thermometer was corrected for in such a way that a radiation source with a small black plate heated at 600°C was located in the atmosphere near the vacuum windows of the chambers, and that it was observed by the multiband radiation thermometer periodically.

2.2. Specimens

Figure 2 shows a standard shape of specimens and a specimen heating assembly. A sheet for a specimen 0.1mm to 0.5mm thick was folded in such a way that the specimen formed a

cavity with an opening slit on the rear. The inner surface of the cavity was coated with heat-resisting black paint (Asahipen, Japan). The cavity was observed at the middle of the opening slit by the single-band radiation thermometer to measure the specimen temperature. Forming a cavity in the specimen as shown in Figure 1 increased the effective emissivity when the specimen was viewed from the rear by the single-band radiation thermometer, and sometimes reduced influence of degradation of the heat-resisting paint.

Figure 2 also shows a specimen holder and a receptacle cooled by water flow. The specimen holder had a flexible lead of bent copper ribbon that connected the bottom of the specimen and the lower electrode to absorb the thermal expansion of the specimen when heated. The specimen was tied to the specimen holder by two screws and nickel bits to make good electric contact. The specimen holder was set to one of the vacuum chambers and the receptacle just by plugging. Several specimen holders of such a plug-type were prepared to improve the measurement efficiency.

A total of thirty kinds of pure metals, steels and alloys were prepared. Specimens were heated up to high temperatures by direct current pass in the vacuum chambers. A power source of 500A with a current regulator was used under manual control.

2.3. Specimen heating and environmental control

Figure 3 shows a front view of the specimen heating and environmental control system. It had two vacuum chambers of the same structure; one was used for pre-heating of specimens and the other for emissivity measurements. The pre-heating is necessary to keep the vacuum window for measurements clean. The two vacuum chambers improved very much the measurement efficiency to accommodate to a large number of specimens.

The chambers were evacuated by a turbo-molecular pump. The air or argon gas with an oxygen density from 100ppm to 10% was introduced into the vacuum chamber 7 liters in volume for oxidation control of the specimen surface. The flow rate of oxidizing gas was regulated by a mass flow controller. The inner surfaces of the vacuum chambers were coated with the heat-resisting black paint to reduce reflection.

2.4. Multiband radiation thermometer

Figure 4 shows the optical system of the multiband radiation thermometer with 112 wavelengths. It consisted of an objective optics with a pointing laser, a 64-element silicon/germanium hybrid linear array detector with a holographic grating ranging from 0.5µm to 1.6µm and a 48-element InSb linear array detector with a LiF prism ranging from 1.3µm to 5.3µm. The use of linear array detectors eliminated mechanisms for grating and prism rotating. Reduction of background thermal radiation onto the InSb detector by using a large-size cold radiation shield contributed stabilization of the zero level of the multiband radiation thermometer that eliminated a mechanical chopper for incident radiation.

The electric circuit consisted of 112 first-stage amplifiers, 8 FET multiplexers, 7 gain-adjuster amplifiers, a 12bit A/D converter, and a personal computer. The optical system and the electric circuit realized a repetitive measurement time as short as 256µs to scan a total of 112 wavelengths.

The multiband radiation thermometer was operated with a period of 0.5s or 1s to reduce the amount of output data in actual measurements considering that the period was short enough to follow the emissivity and temperature changes. Ten A/D conversions for the integration and an automatic gain adjustment of the analog signal were made to each channel of 112 wavelengths every 0.5s.

2.5. Measurement procedures

Before the normal spectral emissivity measurement, the single-band and multiband

radiation thermometers were radiometrically calibrated against a standard radiation thermometer with the operating wavelength of 0.9µm that had an accurate temperature scale established by fixed-point blackbody calibrations. In the radiometric calibrations a radiation source for atmospheric absorption correction and two variable-temperature blackbody furnaces were employed. The wavelength calibration of the multiband radiation thermometer was made by using 24 narrow-band-pass filters.

The surface roughness of the specimen front surface was measured at the middle of each specimen by a contact-type prove after cleaning the front surface with methanol. Then the specimen was tied to the specimen holder. Since the specimen had volatile matter and the heat-resisting paint required hardening, it was slowly pre-heated up to 600°C in one of the vacuum chambers. When the normal spectral emissivity was measured in a vacuum, the specimen was heated in the other vacuum chamber, and then the temperature was varied. When the normal spectral emissivity was measured in an oxidizing environment, the specimen was heated to a certain temperature in the vacuum chamber for measurements, and then a valve of the vacuum pump was closed and an oxidizing gas was introduced.

3. Results and evaluation

3.1. Effective emissivity of specimen cavity

The effective normal spectral emissivity of the specimen rear side was considerably increased by forming a cavity. However it could not be regarded yet as a perfect blackbody. Hence the specimen temperature was corrected for the effective emissivity.

The effective normal spectral emissivity of the specimen cavity was measured by comparison with that of a metal tube having a small hole at the middle. The metal tube was made by welding from a sheet of cold-rolled steel. All specimens used for the comparison were made from the same sheet as the metal tube. The front surfaces of the specimens and the metal tube, and the inner surfaces of the specimen cavities and the metal tube were coated with the same heat-resisting black paint.

The measurement of the effective normal spectral emissivity was made as follows. First, a specimen was heated at a high temperature. Then the radiance temperature of the specimen cavity was measured, and the spectral radiance of the painted front surface was monitored by the multiband radiation thermometer at the wavelength of 2.13µm where the radiance resolution was good enough. Second, the metal tube was heated at the same temperature as the specimen so that the monitoring multiband radiation thermometer indicated the same level of the spectral radiance of the painted front surface of the metal tube. The oxidizing environment and the temperature history were also set in the same way as the specimen. Then the radiance temperature of the small hole was measured by the single-band radiation thermometer. Although a small difference between the radiance temperatures of the specimen and the metal tube remained, it was corrected.

The normal spectral emissivity of the specimen cavities was derived from the calculation assuming that the effective emissivity of the small hole formed on the metal tube was unity and that the emissivity of the painted front surface of the metal tube was the same as that of the painted front surfaces of the specimens. Figure 5 shows effective normal spectral emissivity of the specimen cavities in the course of measurement time that was measured at the middle of the specimen rear slit at the wavelength of $0.9\mu m$ in three different environmental conditions; see the figure caption. The effective normal spectral emissivity of the cavities ranged from 0.91 to 0.99.

Temperature dependence of the effective normal spectral emissivity was also measured in the range from 780°C to 1100°C, and it could not be measured at temperatures above

1100°C because the cold-rolled specimens deformed and the paint came off the metal surfaces. The effective normal spectral emissivity ranged between the minimum of 0.93 and the maximum of 0.97.

3.2. Uncertainty of emissivity measurements

Table I shows uncertainty allocations of the emissivity measurement by the system. Uncertainties in the temperature scales include that in the standard radiation thermometer and the emissivities of the blackbody furnaces used for the calibration. In the short wavelength range from $0.5\mu m$ to $1.5\mu m$, the uncertainty of the effective emissivity of the specimen cavity was the largest one except low temperature measurements and low emissivity specimens. On the other hand, it was not dominant in the long wavelength range.

The total uncertainty was estimated to be 5% or larger of the emissivity value depending on the emissivity, temperature, and wavelength. The lower temperature limit of the measurement and the total uncertainty at the short wavelength range were determined by noise levels of the multiband radiation thermometer; the SN ratio at $0.55\mu m$ was 18 and that at $0.66\mu m$ was 96 under the condition that the target was a blackbody furnace at $1052^{\circ}C$ and the time constant of the electric circuit was $100\mu s$.

3.3. Examples of emissivity data

Figure 7 shows a time variation in the normal spectral emissivity of a Inconel 625 specimen in an oxidation process at 1000°C. A valve of the vacuum pump was closed at the time of zero, and then the air was introduced into the vacuum chamber. The average surface roughness in the vertical direction was 0.12µm. It is seen that the normal spectral emissivity change in a systematic way as oxidation progresses. The specimen color was changed from metallic to black after the measurement.

Figure 8 shows a time variation in the normal spectral emissivity of a Monel specimen that had been oxidized in the atmosphere at about 1000°C before the emissivity measurement. In the emissivity measurement, the specimen temperature was increased from 773°C (a) to 1157°C (b) and then decreased to 998°C (c) in a vacuum. The average surface roughness in the vertical direction was $0.43\mu\text{m}$. While the specimen color was gray before the measurement, it changed to metallic after that.

4. CONCLUSIONS

Time variations in the normal spectral emissivity of metal surfaces in oxidation processes were observed by the emissivity measurement with a sufficiently small time interval. Data throughput of the measurement system was such that one measurement could be finished in an hour including the setting time of the specimen.

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Table I. Uncertainty allocations of the emissivity measurement by the system.

Wavelength	0.9µm	0.9µm	3.7µm	3.7µm
Emissivity	0.1	0.9	0.1	0.9
	Uncertainty (% of the emissivity value)			
Temperature scale of single-band thermometer	1	1	0.2	0.2
" of multiband thermometer	1.5	1.5	2	2
Drift and noise of multiband thermometer	4	3	3	3
Effective emissivity of specimen cavity	5	5	1.5	1.5
Reflection at inner surfaces of vacuum chamber	5	0.5	5	0.5
Absorption of vacuum window and optical pass	2	2	3	3
Total uncertainty	8.4	6.4	7.0	4.9

FIGURE CAPTIONS

- Fig. 1. Schematic of measurement system.
- Fig. 2. Standard shape of specimen, specimen holder, and heating assembly.
- Fig. 3. Front view of specimen heating and environmental control system.
- Fig. 4. Optical system of multiband radiation thermometer.
- Fig. 5. Effective normal spectral emissivity of specimen cavities at the wavelength of 0.9μm. The specimens were painted sheets of cold-rolled steel. (a) the specimen was heated at 1000°C in a vacuum, (b) the temperature was kept at 800°C and the air was introduced into the vacuum chamber at the constant flow rate of 1cc·min⁻¹, (c) the temperature was kept at 1000°C and the air was introduced at the constant flow rate of 10cc·min⁻¹.
- Fig. 6. Time variation in the normal spectral emissivity of a Inconel625 specimen kept at 1000°C when the air was introduced into the vacuum chamber at the constant flow rate of 1cc·min⁻¹ (from 0s to 500s), and then 10cc·min⁻¹ (from 500s to 1000s).
- Fig. 7. Time variation in the normal spectral emissivity of a Monel specimen when varying the temperature in a vacuum. The specimen was oxidized by heating at about 1000°C in the atmosphere before the measurement. (a) the beginning of the measurement, (b) intermediate at the maximum temperature, (c) the measurement end.













